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Measure of Fly Ash Resistivity Using Simulated Flue Gas Environments

Southern Research Inst, Birmingham, Ala

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Research Triangle Park, North Carolina 27711

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MEASUREMENT OF FLY ASH RESISTIVITY USING SIMULATED FLUE GAS ENVIRONMENTS

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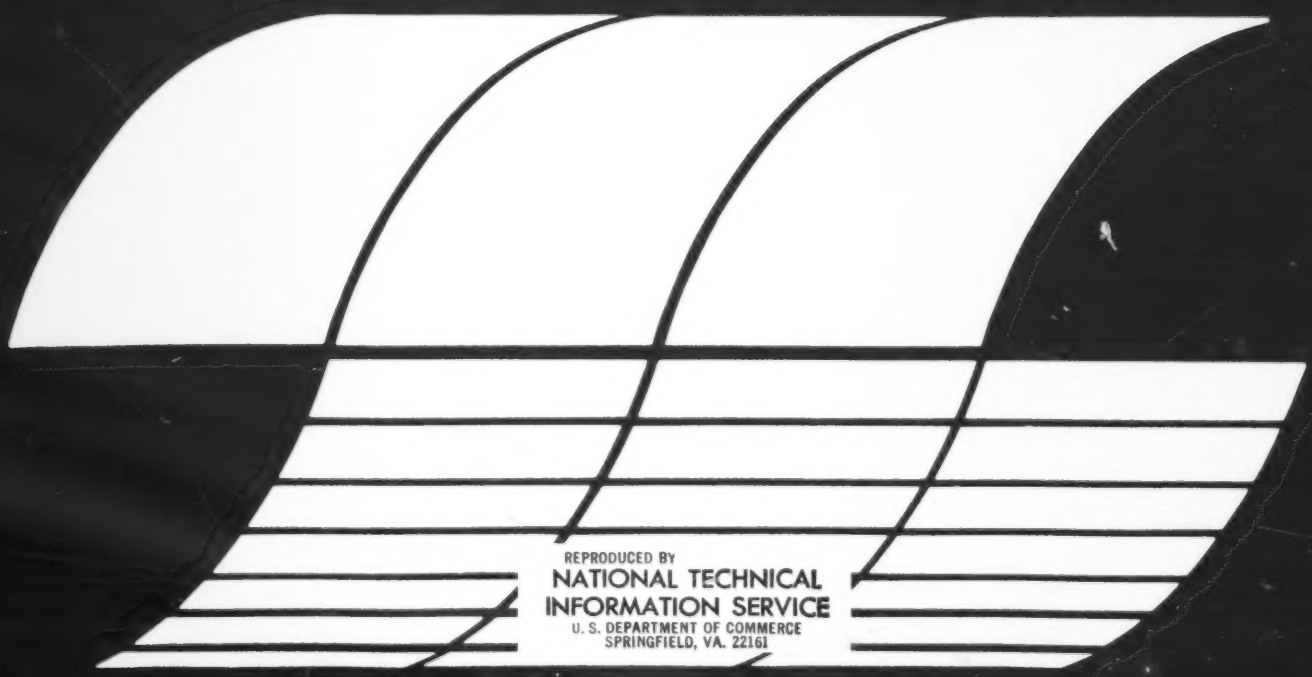
Program Report



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by

R. E. Bickelhaupt

Southern Research Institute
2000 Ninth Avenue, South
Birmingham, Alabama 35205

Contract No. 68-02-2114
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EPA Project Officer: Leslie E. Sparks

Industrial Environmental Research Laboratory
Office of Energy, Minerals and Industry
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ABSTRACT

This report, describing the apparatus and laboratory procedures used to determine resistivity for a number of fly ashes under a variety of test conditions, supports research to develop a technique for predicting fly ash resistivity from chemical analyses of coal and coal ash. This effort requires considerable knowlege regarding the relationship between resistivity and several coal and ash properties. In particular, the report relates the experimental problems encountered when attempts were made to determine the effect of sulfur trioxide on resistivity. Equipment and procedures were developed to solve this problem. The report describes the modified apparatus and technique and illustrates the type of data acquired.

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SECTION I

SUMMARY

The equipment and technique used to measure the resistivity of fly ash in the laboratory at the start of this research program would be considered conventional and adequate according to the general literature. However when SO_x was included in the simulated flue gas, a number of problems occurred that made the existing test apparatus and procedures unsuitable.

Retrospectively reviewed, the principal problems encountered were: a) the presence of uncontrolled amounts of SO_3 due to the catalytic oxidiation of SO_2 when using environments containing SO_2 and O_2, b) the failure to use an adequate total flow of gas to maintain a constant concentration of SO_3 during a test, and c) failure to anticipate the time required to "equilibrate" a rather small quantity of ash in direct contact with an environment containing sulfuric acid vapor.

To overcome the problem regarding the generation of SO_3 within the system, all stainless steel equipment, for which it was technically feasible, was converted to glass, and finally the environment was changed from a mixture of SO_3, SO_2, O_2, H_2O, CO_2 and N_2 to a simple mixture of air, water and SO_3. Changes made to create a system that produced a constant concentration of SO_3 at the test site include: a) converting to a glass system to reduce adsorption by the system, b) increasing the environmental total flow rate at standard conditions from 1.3 to 5.0 liters/minute, and c) reducing the number of ash specimens per test from 4 to 1, and reducing the ash sample area from about 180 to 45 cm^2.

Several steps were taken to develop a test procedure utilizing a sufficient and reasonable period of time to observe the effect of SO_3 (H_2SO_4) on resistivity. It was obvious that the procedure involving varying test temperature would have to be changed to an isothermal test. The conventional parallel plate, ASME PTC-28 test cell was converted to a radial flow cell in which only a surface layer of ash 1 mm thick was under test. The test procedure was altered to allow overnight exposure of the ash surface to the environment. The asymptotic approach of the decreasing resistivity value to some limiting level as a function of ash-environmental exposure time was used to establish a test end point. For many conditions, an exposure lasting nineteen hours was sufficient.

The nature of this investigation, a side excursion into the area of test procedure to be able to accomplish the principal task, precluded a formal approach to the problem. Many small problems have not been discussed, and many trial and error experiments were run. It is difficult therefore to determine whether all changes in equipment and technique were mandatory. In any event, it was concluded that one can determine the effect of SO_3 on the resistivity of fly ash in the laboratory if a known, invariant concentration of SO_3 (H_2SO_4) can be maintained at the test site and if sufficient time is allowed to "equilibrate" the volume of ash under test with the environment.

Time has not permitted the detailed evaluation of the apparatus and procedures described herein. The system was considered suitable for use in continuing the effort regarding the principal research objective, which was the development of a technique for predicting the electrical resistivity of fly ash.

SECTION II

INTRODUCTION

The objective of EPA Contract No. 68-02-2112, Task IV, is to develop a method for predicting fly ash resistivity as a function of temperature from the analyses of coal and coal ashes prepared by simple laboratory ignition. The method must include expressions to reflect the effect of electric field strength, water concentration, sulfur trioxide concentration and ash composition on the resistivity-temperature relationship. To accomplish this, the resistivity-temperature characteristics for a large number of fly ashes were investigated using the above test variables.

The research progressed at a satisfactory pace with encouraging interim results until tests were begun to demonstrate the effect of variations in SO_2 and SO_3 concentrations on ash resistivity as a function of temperature. The purpose of this report is to point out the problems encountered in this facet of the work and to illustrate the equipment and test procedure eventually selected to complete the program.

SECTION III

INITIAL EXPERIMENTAL APPROACH AND PROBLEMS ENCOUNTERED

Before attempting to deal with the difficulties encountered in simulating flue gases containing SO_x, it would be beneficial to define the apparatus and test procedure originally designed for this research. The equipment and technique described below were used successfully prior to studies in which the environment included SO_x.

Experimental Apparatus

The original experimental arrangement was designed to determine resistivity for four ash specimens simultaneously using ASME, PTC-28 test cells[1]. The test cells were contained in a 316 stainless steel chamber that was housed in a high temperature oven. Simulated flue gas environments were maintained in the test chamber under a small positive pressure (1 to 2 inches of water). The electrical circuit allowed the cells to be independently energized for resistivity measurements.

Figure 1 illustrates the physical arrangement of the apparatus. Tank gases including commercially prepared and certified 1% SO_2 in N_2 were metered using precision rotameters to deliver the desired mixture at a total flow rate of 1.3 liters/minute at standard conditions. Depending on the temperature, this flow rate provided 5 to 10 volume changes per hour for the test chamber. The standard or baseline simulated environment contained by volume 5% O_2, 13% CO_2, 9% H_2O, 500 ppm SO_2 and the balance N_2.

The gases leaving the rotameters passed through a stainless steel manifold into a two liter stainless steel mixing vessel held at 200°C to preheat the gas. At the exit of this vessel an inlet was provided for the introduction of SO_3. The proper amount of SO_3 to be injected was governed by the temperature of the 20% sulfuric acid bath and the flow rate of the nitrogen used as a carrier.

A temperature of 160°C was maintained for the stainless steel tubing carrying the gas mixture to the oven. After entering the oven, the gas was passed through 25 feet of tubing, maintained at the test temperature, before it entered the resistivity chamber. Gas exiting the chamber was passed through a bubbler external to

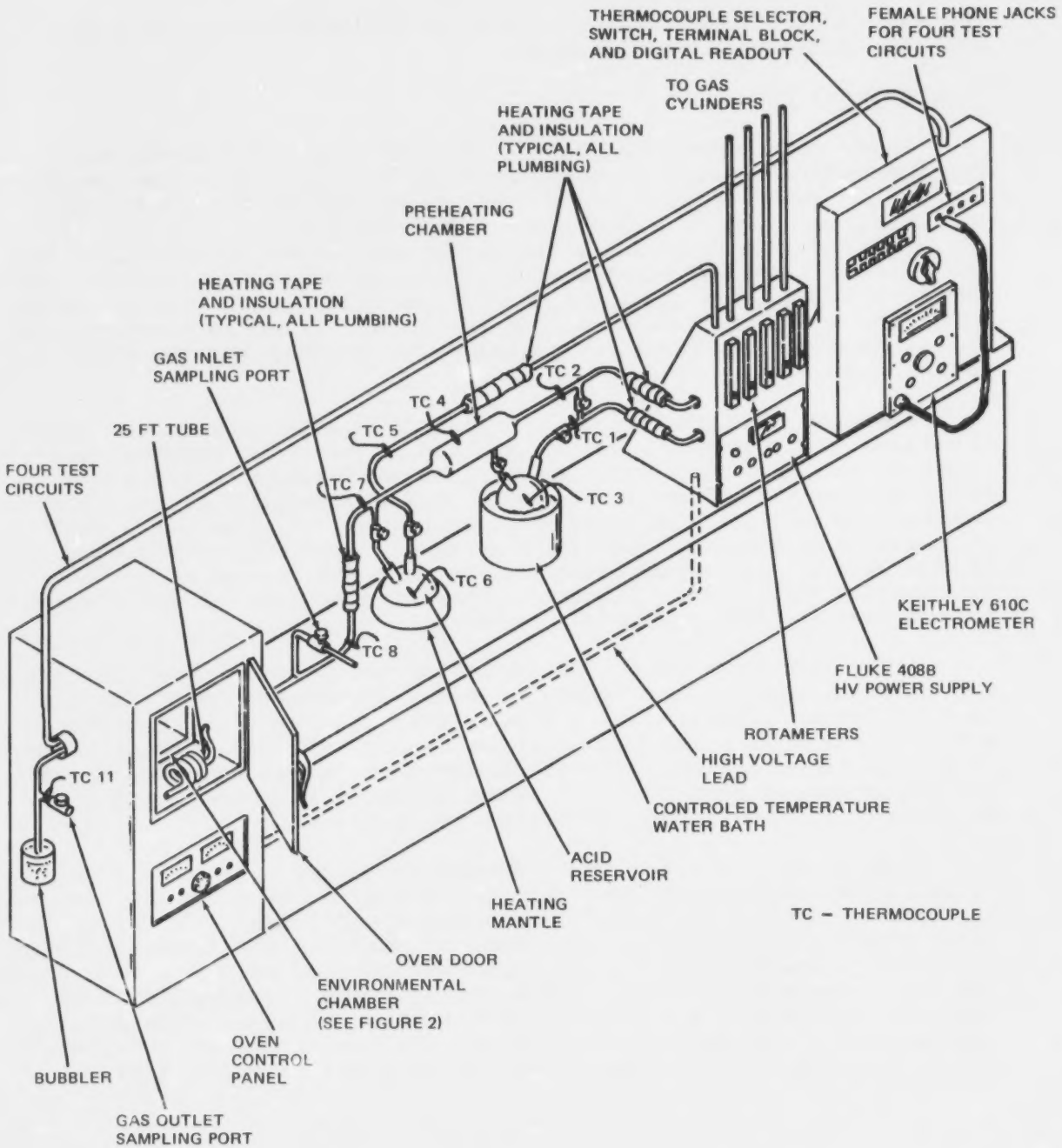


Figure 1. General apparatus arrangement for resistivity measurement initial equipment: chamber and all plumbing 316 stainless steel.

the oven to provide visual evidence of the maintenance of a small positive pressure in the chamber.

Experimental Procedure

Ashes were passed through an 80 mesh screen to remove any foreign material prior to being poured into the cup of the resistivity cell. While being filled, the cup was tapped to insure that ash bridging would be minimized. After the cell surface was leveled, the upper, current-measuring electrode was placed in position, and the test cell was attached to the proper leads in the chamber, see Figure 2. The front piece of the chamber was sealed with C clamps after the four test cells were in position. Clamping together two finely machined surfaces was suitable for maintaining the small internal chamber pressure.

Nitrogen, passed through a drying column and the heated plumbing leading to the oven, was maintained in the test chamber overnight as the specimens were thermally equilibrated at 450-470°C. Prior to converting the environment to a simulated flue gas, the resistivity of each test cell was determined. Sequentially each cell was tested by applying 1000 volts DC (5mm ash layer giving an E = 2kV/cm) and determining the current one minute after the application of voltage. Figure 3 shows a schematic of the electrical circuit for the resistivity test.

After the environment was converted to the simulated flue gas, the current readings were repeated every 10 minutes until the current no longer increased with time. This usually took 20 to 40 minutes. At this point the oven was turned off, and current readings were taken periodically as the chamber temperature decreased. The cells cooled from 460°C to 145°C in about four hours and cooled further to 85°C in an additional two hours.

When it was of interest to determine resistivity as a function of ash layer field strength, the decreasing temperature was arrested at 162°C while the necessary measurements were made. Variation in water concentration was accomplished by changing the temperature of the water through which the nitrogen was bubbled prior to entering the 200°C preheating vessel. The nitrogen was valved so that it could be introduced dry or through the water bubbler. The water concentration was determined from an exit gas sample at least once during each resistivity test. Resistivity was calculated as follows:

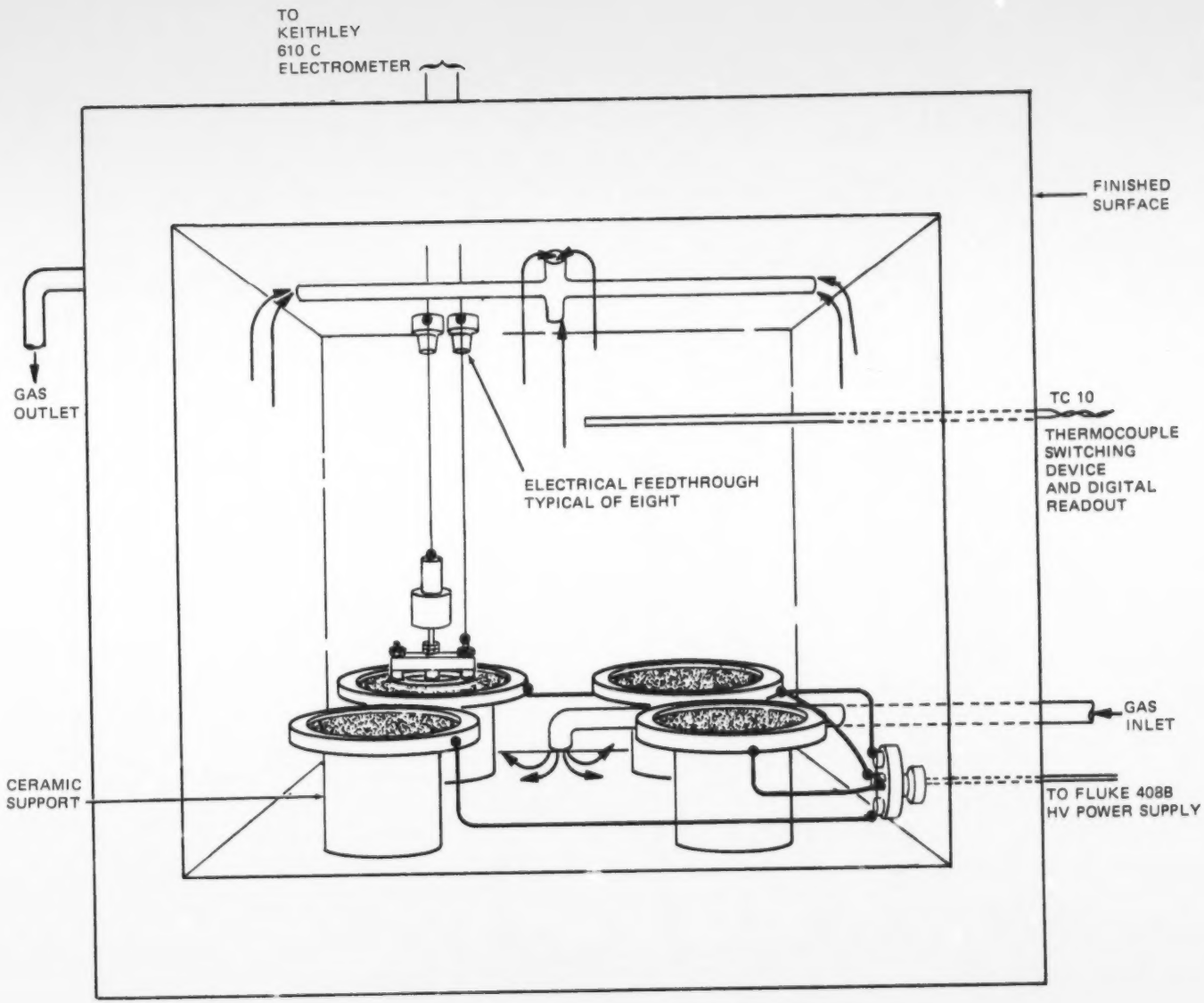


Figure 2. 316 stainless steel environmental resistivity chamber.

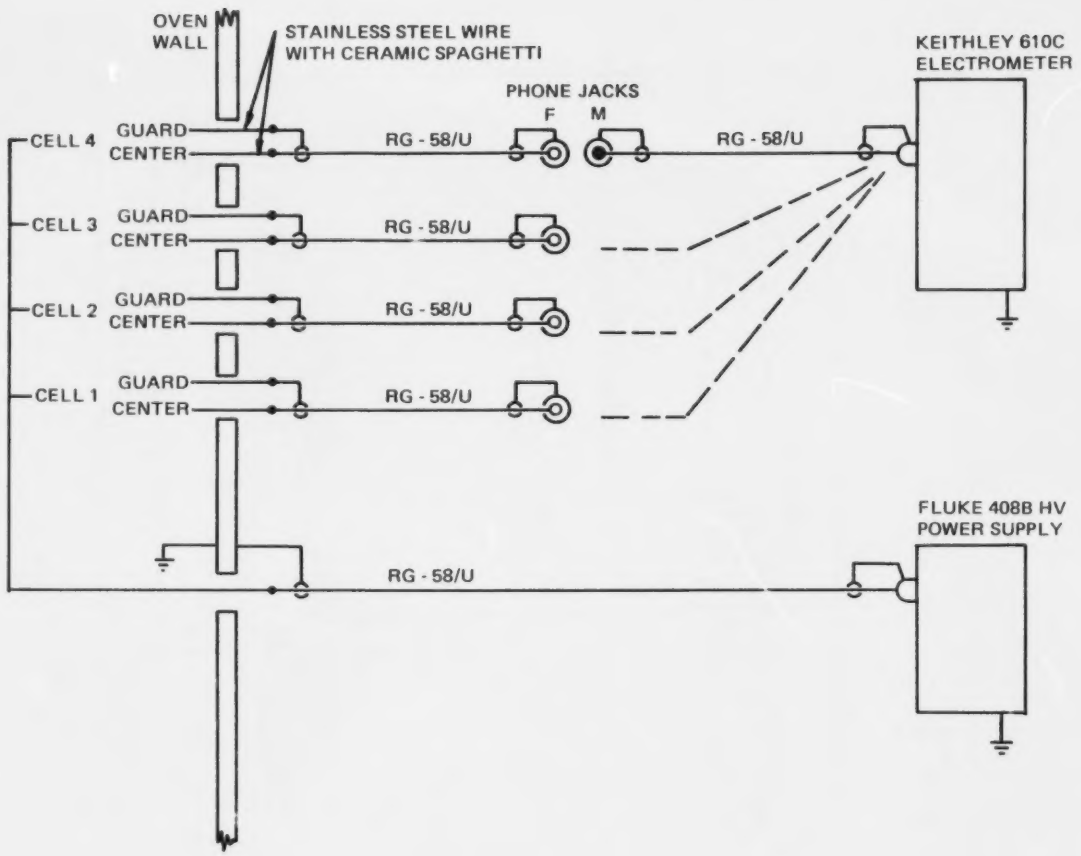


Figure 3. Resistivity test, wiring diagram.

$$\rho = \frac{V \cdot A}{I \cdot L} \qquad (1),$$

where:

ρ = resistivity in ohm·cm

V = D.C. volts

I = current in amperes

A = area of measuring electrode in cm^2

L = thickness of ash layer in cm.

Problems Encountered Using SO_x

It was stated above that the standard or baseline environment contained ∿500 ppm of SO_2 and no injected SO_3. Preliminary experiments had shown a small difference between resistivity data acquired using air-water environments versus the baseline simulated environment. At the time, it was believed that the small attenuation of resistivity was possibly due to the presence of SO_2. (Retrospectively, the SO_2 would have been deleted from the baseline environment if it had been realized that an uncontrolled amount of SO_3 was being introduced by the catalytic oxidation of SO_2.)

The scope of research required the investigation of the effect of simulated environments containing 500, 1000, 2000 and 3000 ppm of SO_2. When the larger concentrations of SO_2 were incorporated, it was observed that resistivity values were significantly attenuated. Although one could not rule out the possibility that SO_2 affects ash resistivity, it seemed likely that large quantities of SO_3 were being generated and that the reduction in resistivity was due to the presence of sulfuric acid. Determination of SO_3 and SO_2 concentrations in the inlet and outlet gas samples when no SO_3 was being injected verified the presence of SO_3. Gas analysis procedure is described in Reference 2.

Several months were spent running ancillary experiments attempting to understand the problem and develop a way in which the existing equipment and test procedure could be utilized. When SO_2 was included in the environment, SO_3 was catalytically produced.

A few ppm were produced even when oxygen was excluded. It was concluded that some oxygen was present as a trace impurity in other gases or that air diffused into the test chamber at the imperfect seal on the face. Furthermore, the amount of SO_3 catalytically produced was sensitive to the plumbing temperature and the temperature of the test chamber. When SO_2 was eliminated and SO_3 was injected, the difference in SO_3 concentration in the inlet and exhaust gas samples from the test chamber was sensitive to the chamber temperature. This indicated the chamber was capable of adsorbing a significant quantity of available SO_3 (H_2SO_4). Since temperature was one of the test variables and since it was desired to keep the SO_3 concentration constant during a specific test, the above observations indicate that the original procedure and equipment were not satisfactory for the evaluation of the effect of SO_3 on resistivity.

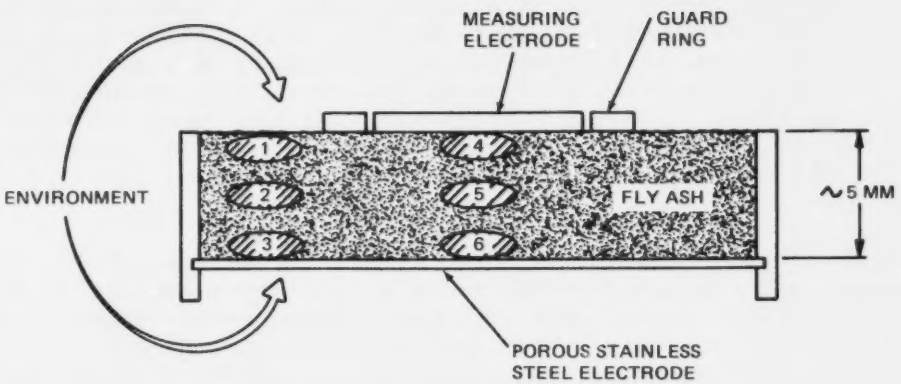
SECTION IV

EXPERIMENTS TO DEVELOP APPARATUS AND PROCEDURE TO UTILIZE ENVIRONMENTS CONTAINING SO_x

A series of modifications took place in reaction to the observed test results. The first modification converted all plumbing and hardware from stainless steel to glass with the exceptions of; electrical feedthroughs, test cells, lead wires, etc. This did not eliminate the formation of SO_3 from the SO_2 and O_2 present in the environment; however, the amount of SO_3 adsorbed by the system was decreased. It was then decided to convert to an environment of air, water vapor and injected SO_3 since no evidence was available to suggest a need for O_2, CO_2 and SO_2 to be present.

Under these conditions, the effect of 10 ppm of SO_3 on resistivity was not observed although a significant amount of SO_3 was removed from the environment as indicated by the measured SO_3 concentrations for chamber inlet and outlet gas samples. [This is in contrast to the observed reduction of resistivity reported for the stainless steel system. It has been rationalized that in the case of the earlier observations either a very great quantity of SO_3 had been generated and/or condensation of acid had taken place.] At this point the total environmental flow rate under standard conditions was increased from 1.3 liters/minute to 5.0 liters/minute, and the number of test cells were reduced from four to one. Under these conditions and with 25 grams of ash present in the single test cell, an injection rate of $\sim$10 ppm SO_3 could be maintained in both the inlet and outlet gas samples.

However, even overnight exposure to an environment consisting of air containing 9% water and 10 ppm of SO_3 did not produce a significant attenuation of resistivity. The resistivity cell was the type suggested in ASME PTC-28, previously referenced. The ash is held in a shallow dish having a porous, stainless steel bottom. The upper ash surface is exposed to the environment except where the measuring electrode and guard ring rest. Ash specimens were taken at various elevations between the exposed surface and the porous metal base at plan positions exposed to the environment and beneath the measuring electrode. The amount of soluble sulfate was determined[2] for each specimen as a measure of the penetration and adsorption of sulfuric acid from the environment. The results are shown in Figure 4 for an ash having a soluble sulfate value of



SCHEMATIC CROSS-SECTION OF ASME, PTC-28 RESISTIVITY CELL

SAMPLE POSITION:	BLANK	1	2	3	4	5	6
% SOLUBLE SULFATE:	0.20/0.25	0.80	0.41	0.34	0.25	0.28	0.28

Figure 4. Weight percent soluble sulfate.

0.20 - 0.25% before testing.

These data show that even after 24 hours of exposure at 145°C to an environment consisting of air, 9% water and 10 ppm of SO_3, a large concentration gradient of adsorbed acid (soluble sulfate) exists through the ash layer. The data show that in the area directly exposed to the environment the acid pickup was significant at the surface and a concentration gradient developed from position 1 to 3. Between the measuring electrodes there was little adsorption of acid. Therefore, no appreciable attenuation of resistivity was noted. Obviously even a thin ash layer (1-2mm) between two parallel, porous electrodes would not be a successful test geometry under these conditions.

Attempts to utilize vacuum to pull the environment through the electrodes and ash layer and other schemes to force it through under pressure failed. Besides the side effects of either compacting or fluidizing the ash layer, the concentration gradient of acid pick-up expressed as soluble sulfate could not be eliminated.

The observations described above suggest that in addition to the ASME resistivity cell, other designs may be unsatisfactory for examining the effect of SO_3 on resistivity. Nevens et al[3] recently evaluated three general types of laboratory resistivity test cells. Since these cells require the environment to permeate a porous stainless steel electrode and about 5 mm of ash, these designs are probably undesirable for environments involving SO_x.

Kanowski and Coughlin[4] were successful in illustrating the evfect of SO_3 on fly ash resistivity using a cell believed to be similar to that suggested by ASME-PTC-28. Although all apparatus and procedural details are not available, it would seem that the use of very high total environmental flow rates and the use of high concentrations ($\sim$30ppm) of SO_3 contributed to this success. This approach was not attempted in the subject research, because the facilities limited the low rates available and interest was restricted to low SO_3 concentrations, <10ppm.

SECTION V

DEVELOPMENT OF A RADIAL FLOW TEST CELL AND PROCEDURE

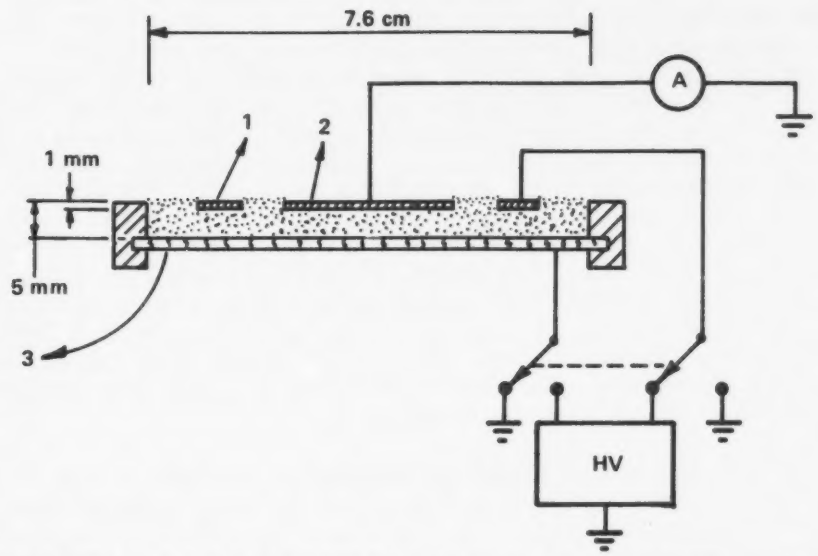
Equipment

The observation that the exposed ash surface adsorbed a significant amount of sulfuric acid (soluble sulfate), and the assumption that a thin layer of ash at the surface must become essentially "equilibrated" with the environment in a reasonable period of time lead to the development of a test apparatus and technique that has provided useful laboratory resistivity data. Initial experiments showed that surface resistance readily reflected the effect of sulfuric acid in the environment. The test cell shown in Figure 5 was constructed to compare simultaneously a conventional test cell with a radial flow test cell using a 1 mm thick ash layer. With this arrangement, one can alternately measure resistivity in the conventional parallel plate mode between electrodes 2 and 3 or in the radial flow mode between electrodes 1 and 2. The cell dimensions selected were based on the work of Amey and Hamburger[5] regarding optimum geometries for surface and volume resistance measurements. Resistivity can be calculated for the radial flow cell from the expression:

$$\rho = \frac{2\,\pi\,c}{\ell n\,(r_2/r_1)} \cdot \frac{V}{I} = \frac{1.56V}{I} \qquad (2),$$

where:

- ρ - resistivity, ohm·cm
- V - volts, applied between electrodes 1 and 2
- I - amperes, current flowing between electrodes 1 and 2
- c - 0.1cm, thickness of electrodes 1 and 2
- r_2 - 1.90cm, radius of I.D. of electrode 1
- r_1 - 1.27cm, radius of electrode 2



ELECTRODE 1 - 5.1 cm OD x 3.8 cm ID x 0.1 cm THICK, SOLID STAINLESS STEEL
ELECTRODE 2 - 2.54 cm OD x 0.1 cm THICK, SOLID STAINLESS STEEL
ELECTRODE 3 - 7.64 cm OD x 0.1 cm THICK, POROUS STAINLESS STEEL

Figure 5. Combination parallel plate - radial flow resistivity test cell and electrical circuit.

Figure 6 shows a radial flow cell in the glass environmental chamber.

Figure 7 shows the comparative results for the two electrode geometries expressed as resistivity versus time of environmental exposure. For this experiment the apparatus shown in Figures 5 and 6 was used, and the ash was thermally equilibrated overnight in dry air at 145°C. Resistivity was determined, about 1.4×10^{13} ohm·cm with either electrode set, and the environment was changed to include 9% water at time = 0 hours. After 20 minutes, both electrode sets measured a resistivity of 2 to 3×10^{11} ohm·cm. This response time is typical. At this temperature, flow rate and chamber size, the time required to dilute a given environmental composition to 99% of a different composition was about six minutes. After the minimum resistivity due to water injection is reached, the resistivity gradually increases with time of exposure. Even though the injection of 10 ppm of SO_3 was started at time equal 30 minutes, the linear flow, parallel plate electrode set showed this increase in resistivity; i.e., the parallel plate electrode set did not respond to the presence of SO_3. However, the resistivity measured with the radial flow electrode set started to show the effect of SO_3 injection about 30 to 60 minutes after injection was started. After about two hours had elapsed, the attenuation of resistivity due to SO_3 injection was quite apparent and continued at a decreasing rate until a minimum value was attained about 24 hours after the start of the test. For this ash and set of conditions, it is assumed that a 24 hour exposure was required to "equilibrate" the 1 mm thick ash layer between electrodes 1 and 2, Figure 5, with the surrounding environment of air, water vapor and sulfuric acid vapor.

No effort has been made to formally evaluate the reproducibility of data using this cell; however, the cursory comparison of many pairs of tests would indicate the reproducibility is good. Also, no attempt has been made to evaluate the effect of variations in the test procedure on the data generated. It has been noted that the inlet and outlet SO_3 determinations indicate the environment is reproducible and that typically the inlet concentration is slightly greater than the outlet concentration for injections of <10ppm SO_3.

Test Procedure

The following test procedure was used to determine the resistivity for a number of ashes as a function of temperature and SO_3 concentration. This procedure is started at 11 AM each day that a test is to be conducted.

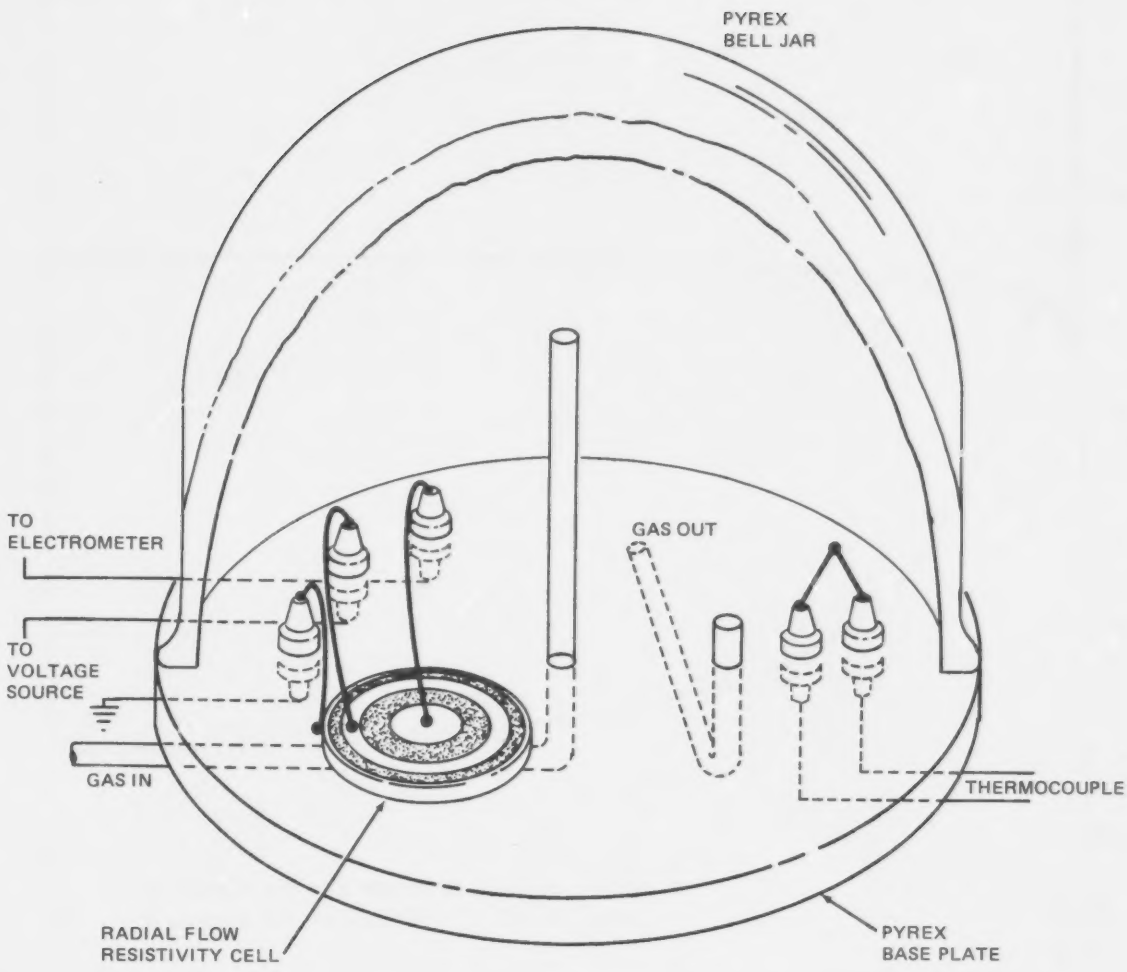


Figure 6. Glass environmental resistivity chamber.

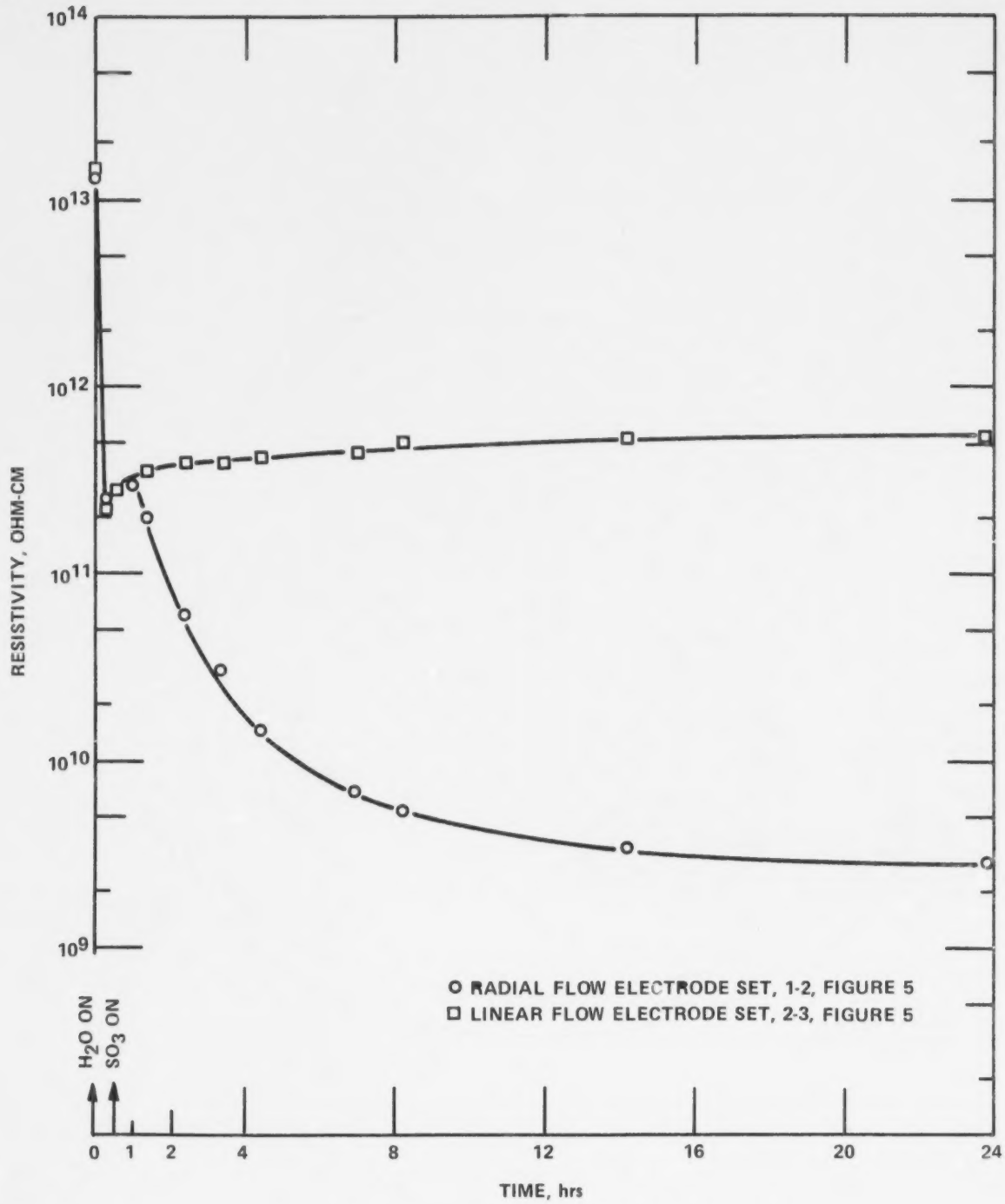


Figure 7. Resistivity vs. time of environmental exposure.

...load cup of resistivity test cell with ash in the manner previously mentioned,

...place cup in chamber,

...attach lead wires and insert electrodes 1 and 2 by pressing them into the ash layer using a straight edge until ash slightly flows on to top of electrodes,

...cover chamber base plate with bell jar, start flow of dry air and turn on oven to desired set point.

...determine hot, dry resistivity at 2 PM and then divert dry air flow through controlled temperature water bubbler to introduce water vapor,

...determine resistivity at 2:15 and 2:30 PM and start nitrogen flow to inject desired concentration of SO_3,

...determine resistivity at 3:30 PM and take inlet and outlet gas samples for SO_3 determination,

...determine resistivity at 4:30 PM,

...determine resistivity at 8:00 AM, take inlet and outlet gas samples for SO_3 determination and outlet gas sample for water concentration,

...determine resistivity at 9:00 AM, convert environment to dry air and cool oven,

...open oven when cool and take ash sample for soluble sulfate determination.

...begin new test at 11 AM.

Resistivity is calculated using equation (2) above. Current is determined one minute after 1200 volts are applied to electrodes 1 and 2 in Figure 5.

The present apparatus has two severe disadvantages: only one test can be run per day and it is possible that in some cases the minimum resistivity could occur during the hours when no one is attending the apparatus or could require environmental exposure greater than 19 hours. It is hoped that in time these shortcomings will be overcome. Since the acquisition of quantitative information regarding the effect of SO_3 on fly ash resistivity was vital to this research, the effort has continued by obtaining data of this type rather than improving or perfecting the test apparatus and technique.

Typical Data

Resistivity was determined at 139, 149 and 173°C using the isothermal test procedure described immediately above. Sulfur trioxide concentrations of 0, 5 and 10 ppm were used in an air environment containing 9 volume percent water. The results are shown in Figure 8 along with the data (circles) for the same ash tested using the procedure described at the beginning of this report. The difference in resistivity shown by circle and square symbols is a reflection of the variation in test procedure. The attenuation of resistivity due to the injection of low to moderate concentrations of sulfur trioxide is dramatic.

The radial flow cell is presently being used to gather additional data similar to those shown in Figure 8, to conduct various miscellaneous experiments to aid in data interpretation, and to elucidate the mechanism by which the sulfuric acid vapor participates in the conduction process. While the radial flow cell and test procedure described have not been extensively evaluated and possess certain disadvantages, both the new cell and test procedure are believed to be worthwhile technical advances.

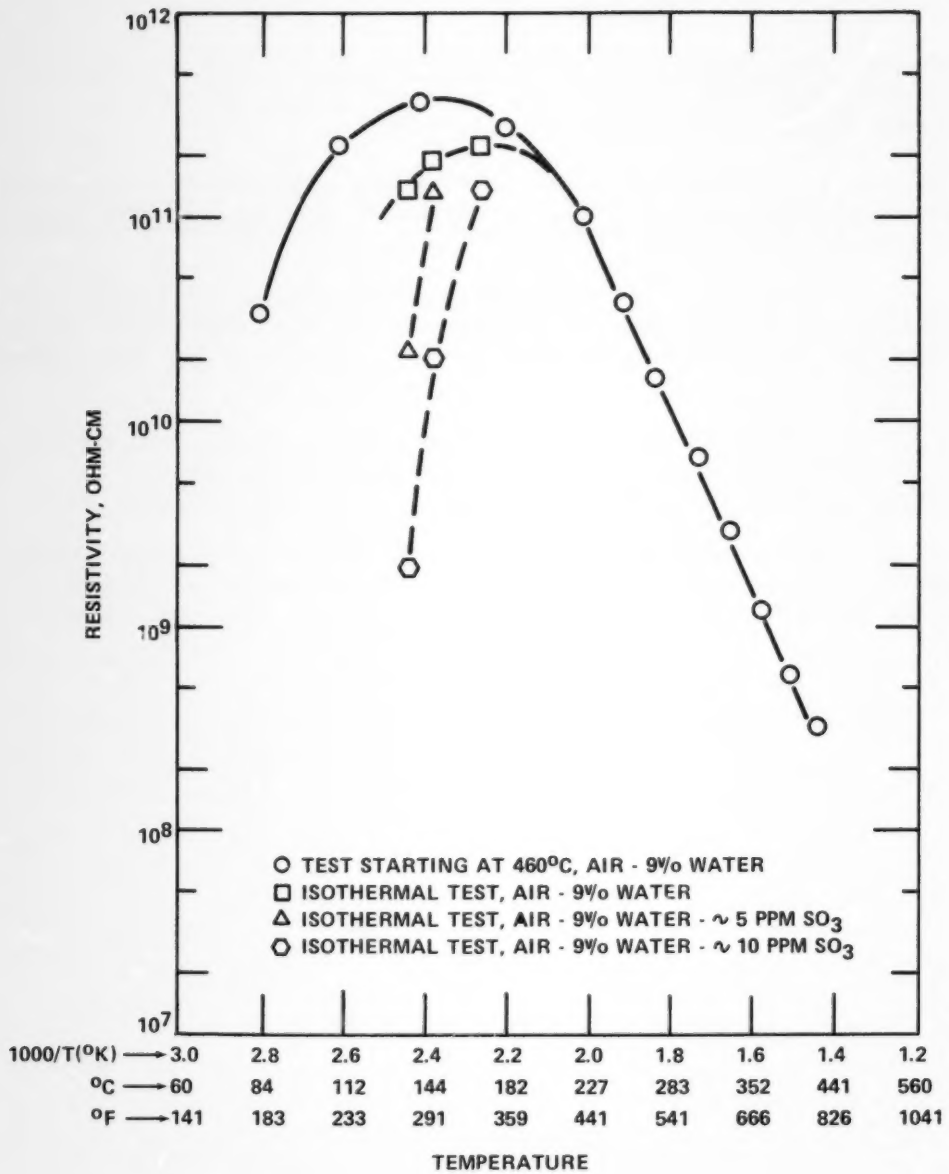

RESISTIVITY, OHM-CM
10^{12}
10^{11}
10^{10}
10^{9}
10^{8}
10^{7}
TEST STARTING AT 460°C, AIR - 9% WATER
ISOTHERMAL TEST, AIR - 9% WATER
ISOTHERMAL TEST, AIR - 9% WATER - ~ 5 PPM SO_3
ISOTHERMAL TEST, AIR - 9% WATER - ~ 10 PPM SO_3
1000/T(°K) → 3.0 2.8 2.6 2.4 2.2 2.0 1.8 1.6 1.4 1.2
°C → 60 84 112 144 182 227 283 352 441 560
°F → 141 183 233 291 359 441 541 666 826 1041
TEMPERATURE

Figure 8. Effect of SO_3 on resistivity.

SECTION VI

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